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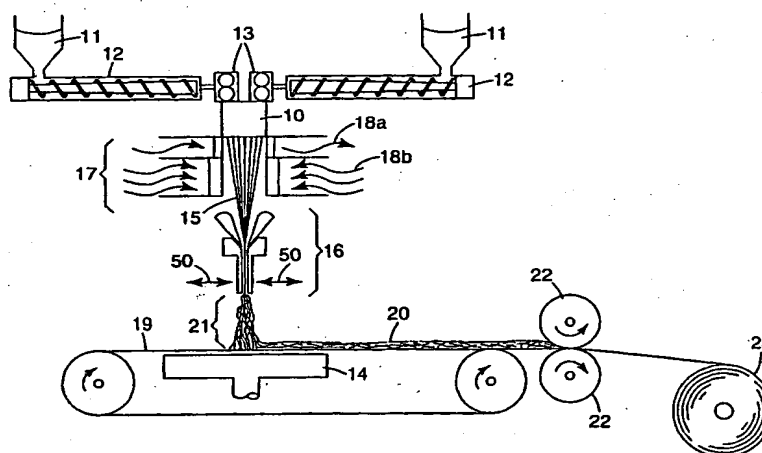
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(54) Title: FIBER-FORMING PROCESS



(57) Abstract: A new fiber-forming method, and related apparatus, are taught in which extruded filaments of fiber-forming material are directed through a processing chamber that is defined by two parallel walls, at least one of which is instantaneously movable toward and away from the other wall; preferably both walls are instantaneously movable toward and away from one another. Movement means are connected to the at least one movable wall to provide instantaneous movement. In one embodiment, the movement means comprises biasing means for resiliently biasing the wall toward the other wall. Movement of the wall toward and away from the other wall is sufficiently easy and rapid that the wall will move away from the other wall in response to increases in pressure within the chamber but will be quickly returned to its original position by the biasing means upon resumption of the original pressure within the chamber. In another embodiment the movement means comprises oscillating means for oscillating the wall at a rapid rate. The invention also provides new nonwoven webs, which comprise a collected mass of fibers that includes fibers randomly interrupted by isolated fiber segments that comprise oriented polymer chains but differ in morphology from the main portion of the fiber.

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FIBER-FORMING PROCESS

In many fiber-forming processes, filamentary material extruded from a die is directed through a processing chamber where, for example, the filamentary material is drawn, oriented and/or reduced in diameter. Such a processing or attenuation chamber is commonly used in spun-bond processes (see U.S. Patent Nos. 3,502,763; 3,692,618; 4,064,605; 4,217,387; 4,812,112; 4,820,459; 5,270,107; 5,292,239; 5,571,537; 5,648,041; and 5,688,468). But it also can be used in other processes, such as meltblowing processes (see U.S. Patent Nos. 4,622,259 and 4,988,560), meltspinning of filaments and filament yarns (see U.S. Patent No. 4,202,855), and flashspinning of plexifilamentary film-fibril material.

Use of a processing chamber places restrictions on the whole fiber-forming process – restrictions intended to assure that fibers will travel through the chamber effectively without, for example, plugging the chamber. Such restrictions include limits on the speed of the fibers as they move through the chamber; limits on the configuration of the chamber to allow threading of fibers through the chamber and rethreading upon breakage of fibers; and limits on the degree to which the extruded fibers are molten or liquid as they enter the chamber.

Various efforts have been made to improve processing chambers and reduce the restrictions they impose on the fiber-forming process. One proposal is to use a wide-throated entry for the chamber, and form the chamber with a movable wall that is moved into place after polymer flow begins and which may be moved out of place if plugging occurs; see U.S. Patent Nos. 4,405,297 and 4,340,563 as well as 4,627,811. Alternatively, U.S. Patent No. 6,136,245 proposes beginning the fiber-forming process slowly and with a processing chamber spaced further from the extrusion die than the intended operating distance; the process is then gradually accelerated and the processing chamber moved closer to the die until it is eventually in the operating position.

In a different effort intended to achieve uniform fiber velocity across the width of an attenuation chamber, the walls of the chamber are made of a flexible material, and a grid of pressure sensors is used to activate local changes in the geometry of the wall to attempt to equalize the pressure through the chamber width; see U.S. Patent No.

5,599,488. U.S. Patent No. 4,300,876 describes a blower structure having only one wall curved to provide a Coanda air stream within which extruded filaments are entrained.

All of these approaches continue to leave important restrictions imposed on fiber-forming processes by use of a processing chamber.

5 The present invention provides a new fiber-forming method that not only alleviates many of the limitations imposed by use of a processing chamber, but more than that, greatly expands fiber-forming and fibrous-web-forming opportunities. In this new fiber-forming method, extruded filaments of fiber-forming material are directed through a processing chamber that is defined by two parallel walls, at least one of which is
10 instantaneously movable toward and away from the other wall; preferably both walls are instantaneously movable toward and away from one another. By "instantaneously movable" it is meant that the movement occurs quickly enough that the fiber-forming process is essentially uninterrupted; e.g., there is no need to stop the process and re-start it. If, for example, a nonwoven web is being collected, collection of the web can continue
15 without stopping the collector, and a substantially uniform web is collected.

 The wall(s) can be moved by a variety of movement means. In one embodiment the at least one movable wall is resiliently biased toward the other wall; and a biasing force is selected that establishes a dynamic equilibrium between the fluid pressure within the chamber and the biasing force. Thus, the wall can move away from the other wall in
20 response to increases in pressure within the chamber, but it is quickly returned to the equilibrium position by the biasing force upon resumption of the original pressure within the chamber. If extruded filamentary material sticks or accumulates on the walls to cause an increased pressure in the chamber, at least one wall rapidly moves away from the other wall to release the accumulated extrudate, whereupon the pressure quickly returns to its
25 original pressure, and the movable wall returns to its original position. Although some brief change in the operating parameters of the process may occur during the movement of the wall(s), no stoppage of the process occurs, but instead fibers continue to be formed and collected.

 In a different embodiment of the invention the movement means is an oscillator
30 that rapidly oscillates the wall(s) between its original position defining the chamber space, and a second position further from the other wall. Oscillation occurs rapidly, causing essentially no interruption of the fiber-forming process, and any extrudate accumulated in

the processing chamber that could plug the chamber is regularly released by the spreading apart of the wall(s).

In general, a new fiber-forming method of the invention comprises a) extruding filaments of fiber-forming material; b) directing the filaments through a processing chamber defined by two parallel walls, at least one of the walls being instantaneously movable toward and away from the other wall and being subject to movement means for causing instantaneous movement during passage of filaments; and c) collecting the processed filaments.

A processing chamber having an instantaneously movable wall as described makes possible great changes in the fiber-forming process. Procedures and parameters that were previously not useful because of the danger of plugging of the processing chamber now become possible. Fiber velocities, polymer flow rates, and degrees to which the polymer is molten or liquid upon entering the processing chamber can be varied to create improved as well as essentially new processes. The invention is especially useful to enhance processes of direct web formation, i.e., processes in which fiber-forming material is directly converted into nonwoven web form, without separate formation of fibers that are then assembled into a web in a different process.

The invention also provides and makes use of a new apparatus, which briefly summarized, comprises a) an extrusion head for extruding filaments of fiber-forming material through orifices in a die, b) a chamber aligned to receive the extruded filaments for passage through the chamber, the chamber being defined by two parallel walls, at least one of the walls being instantaneously movable toward and away from the other wall; and c) movement means for moving the at least one wall, e.g., resiliently biasing the wall toward the other wall or oscillating the wall toward and away from the other wall.

Movement of the wall toward and away from the other wall is sufficiently easy as to allow the described rapid or instantaneous movement, e.g., the wall will move away from the other wall in response to increases in pressure within the chamber but will be quickly returned to its original position by the biasing means upon resumption of the original pressure within the chamber; or the oscillating means will rapidly oscillate the wall between closer and further spacings.

The present invention also provides new products. As discussed in more detail later in this specification, collected masses of fibers from a fiber-forming process of the

invention may include fibers that are interrupted along their length, e.g., by a fiber break or entanglement. The fiber segment where the interruption occurs may differ from the main portion of the fiber in important properties, e.g., in morphological characteristics that are manifested as differences in melting point, cold-crystallization temperature, glass transition temperature, crystallinity index (indicating the proportion of the fiber that is crystalline), or crystalline type. These differences can be detected by differential scanning calorimetry or X-ray scattering. Collected masses of fibers as described are a consequence of the beneficial new fiber-forming process of the invention; and in addition, the new webs offer beneficial properties themselves. One such useful product of the invention comprises a coherent mass of fibers in web form, the mass of fibers including fibers randomly interrupted along their length by segments that are fiber-like, and are less than 300 micrometers in diameter but larger in diameter than the main portion of the fiber.

Figure 1 is a schematic overall diagram of apparatus of the invention for forming a nonwoven fibrous web.

Figure 2 is an enlarged side view of a processing chamber useful in the invention, with mounting means for the chamber not shown.

Figure 3 is a top view, partially schematic, of the processing chamber shown in Figure 2 together with mounting and other associated apparatus.

Figure 4 is a scanning electron micrograph of a web prepared in Example 5.

Figures 5, 6, 7 and 7a are plots obtained by differential scanning calorimetry on various exemplary webs of the invention.

Figure 1 shows an illustrative apparatus for carrying out the invention. Fiber-forming material is brought to an extrusion head 10 -- in this illustrative apparatus, by introducing a fiber-forming material into hoppers 11, melting the material in an extruder 12, and pumping the molten material into the extrusion head 10 through a pump 13. Although solid polymeric material in pellet or other particulate form is most commonly used and melted to a liquid, pumpable state, other fiber-forming liquids such as polymer solutions could also be used.

The extrusion head 10 may be a conventional spinnerette or spin pack, generally including multiple orifices arranged in a regular pattern, e.g., straightline rows. Filaments 15 of fiber-forming liquid are extruded from the extrusion head and conveyed to a processing chamber or attenuator 16. The distance 17 the extruded filaments 15 travel

before reaching the attenuator 16 can vary, as can the conditions to which they are exposed. Typically, quenching streams of air or other gas 18 are presented to the extruded filaments by conventional methods and apparatus to reduce the temperature of the extruded filaments 15. Alternatively, the streams of air or other gas may be heated to facilitate drawing of the fibers. There may be one or more streams of air (or other fluid) - e.g., a first air stream 18a blown transversely to the filament stream, which may remove undesired gaseous materials or fumes released during extrusion; and a second quenching air stream 18b that achieves a major desired temperature reduction. Depending on the process being used or the form of finished product desired, the quenching air may be sufficient to solidify the extruded filaments 15 before they reach the attenuator 16. In other cases the extruded filaments are still in a softened or molten condition when they enter the attenuator. Alternatively, no quenching streams are used; in such a case ambient air or other fluid between the extrusion head 10 and the attenuator 16 may be a medium for any change in the extruded filaments before they enter the attenuator.

The filaments 15 pass through the attenuator 16, as discussed in more detail below, and then exit. Most often, as pictured in Figure 1, they exit onto a collector 19 where they are collected as a mass of fibers 20 that may or may not be coherent and take the form of a handleable web. The invention is particularly useful as a direct-web-formation process in which a fiber-forming polymeric material is converted into a web in one essentially direct operation, such as is done in spunbond or meltblown processes. Alternatively, fibers exiting the attenuator may take the form of monofilaments, tow or yarn, which may be wound onto a storage spool or further processed.

The collector 19 is generally porous and a gas-withdrawal device 14 can be positioned below the collector to assist deposition of fibers onto the collector. The distance 21 between the attenuator exit and the collector may be varied to obtain different effects. The collected mass 20 may be conveyed to other apparatus such as calenders, embossing stations, laminators, cutters and the like; or it may be passed through drive rolls 22 and wound into a storage roll 23. After passing through a processing chamber of the invention, but prior to collection, extruded filaments or fibers may be subjected to a number of additional processing steps not illustrated in Figure 1, e.g., further drawing, spraying, etc.

Figure 2 is an enlarged side view of a representative processing device, namely an attenuator 16, which comprises two movable halves or sides 16a and 16b separated so as

to define between them the processing chamber 24: the facing surfaces of the sides 16a and 16b form the walls of the chamber. Figure 3 is a top and somewhat schematic view at a different scale showing the representative attenuator 16 and some of its mounting and support structure. As seen from the top view in Figure 3, the processing or attenuation chamber 24 is generally an elongated slot, having a transverse length 25 (transverse to the path of travel of filaments through the attenuator), which can vary depending on the number of filaments being processed.

Although existing as two halves or sides, the attenuator functions as one unitary device and will be first discussed in its combined form. (The structure shown in Figures 2 and 3 is representative only, and a variety of different constructions may be used.) The representative attenuator 16 includes slanted entry walls 27, which define an entrance space or throat 24a of the attenuation chamber 24. The entry walls 27 preferably are curved at the entry edge or surface 27a to smooth the entry of air streams carrying the extruded filaments 15. The walls 27 are attached to a main body portion 28, and may be provided with a recessed area 29 to establish a gap 30 between the body portion 28 and wall 27. Air may be introduced into the gaps 30 through conduits 31, creating air knives (represented by the arrows 32) that increase the velocity of the filaments traveling through the attenuator, and that also have a further quenching effect on the filaments. The attenuator body 28 is preferably curved at 28a to smooth the passage of air from the air knife 32 into the passage 24. The angle (α) of the surface 28b of the attenuator body can be selected to determine the desired angle at which the air knife impacts a stream of filaments passing through the attenuator. Instead of being near the entry to the chamber, the air knives may be disposed further within the chamber.

The attenuation chamber 24 may have a uniform gap width (the horizontal distance 33 on the page of Figure 2 between the two attenuator sides is herein called the gap width) over its longitudinal length through the attenuator (the dimension along a longitudinal axis 26 through the attenuation chamber is called the axial length). Alternatively, as illustrated in Figure 2, the gap width may vary along the length of the attenuator chamber. Preferably, the attenuation chamber is narrower internally within the attenuator; e.g., as shown in Figure 2, the gap width 33 at the location of the air knives is the narrowest width, and the attenuation chamber expands in width along its length toward the exit opening 34, e.g., at an angle β . Such a narrowing internally within the attenuation

chamber 24, followed by a broadening, creates a venturi effect that increases the volume of air inducted into the chamber and adds to the velocity of filaments traveling through the chamber. In a different embodiment, the attenuation chamber is defined by straight or flat walls; in such embodiments the spacing between the walls may be constant over their
5 length, or alternatively the walls may slightly diverge (preferred) or converge over the axial length of the attenuation chamber. In all these cases, the walls defining the attenuation chamber are regarded as parallel herein, because the deviation from exact parallelism is relatively slight. As illustrated in Figure 2, the walls defining the main portion of the longitudinal length of the passage 24 may take the form of plates 36 that are
10 separate from, and attached to, the main body portion 28.

The length of the attenuation chamber 24 can be varied to achieve different effects; variation is especially useful with the portion between the air knives 32 and the exit opening 34, sometimes called herein the chute length 35. The angle between the chamber walls and the axis 26 may be wider near the exit 34 to change the distribution of fibers
15 onto the collector; or structure such as deflector surfaces, Coanda curved surfaces, and uneven wall lengths may be used at the exit to achieve a desired spreading or other distribution of fibers. In general, the gap width, chute length, attenuation chamber shape, etc. are chosen in conjunction with the material being processed and the mode of treatment desired to achieve desired effects. For example, longer chute lengths may be useful to
20 increase the crystallinity of prepared fibers. Conditions are chosen and can be widely varied to process the extruded filaments into a desired fiber form.

As illustrated in Figure 3, the two sides 16a and 16b of the representative attenuator 16 are each supported through mounting blocks 37 attached to linear bearings 38 that slide on rods 39. The bearing 38 has a low-friction travel on the rod through
25 means such as axially extending rows of ball-bearings disposed radially around the rod, whereby the sides 16a and 16b can readily move toward and away from one another. The mounting blocks 37 are attached to the attenuator body 28 and a housing 40 through which air from a supply pipe 41 is distributed to the conduits 31 and air knives 32.

In this illustrative embodiment, air cylinders 43a and 43b are connected,
30 respectively, to the attenuator sides 16a and 16b through connecting rods 44 and apply a clamping force pressing the attenuator sides 16a and 16b toward one another. The clamping force is chosen in conjunction with the other operating parameters so as to

balance the pressure existing within the attenuation chamber 24. In other words, the clamping force and the force acting internally within the attenuation chamber to press the attenuator sides apart as a result of the gaseous pressure within the attenuator are in balance or equilibrium under preferred operating conditions. Filamentary material can be extruded, passed through the attenuator and collected as finished fibers while the attenuator parts remain in their established equilibrium or steady-state position and the attenuation chamber or passage 24 remains at its established equilibrium or steady-state gap width.

During operation of the representative apparatus illustrated in Figures 1-3, movement of the attenuator sides or chamber walls generally occurs only when there is a perturbation of the system. Such a perturbation may occur when a filament being processed breaks or tangles with another filament or fiber. Such breaks or tangles are often accompanied by an increase in pressure within the attenuation chamber 24, e.g., because the forward end of the filament coming from the extrusion head or the tangle is enlarged and creates a localized blockage of the chamber 24. The increased pressure is sufficient to force the attenuator sides or chamber walls 16a and 16b to move away from one another. Upon this movement of the chamber walls the end of the incoming filament or the tangle can pass through the attenuator, whereupon the pressure in the attenuation chamber 24 returns to its steady-state value before the perturbation, and the clamping pressure exerted by the air cylinders 43 returns the attenuator sides to their steady-state position. Other perturbations causing an increase in pressure in the attenuation chamber include "drips," i.e., globular liquid pieces of fiber-forming material falling from the exit of the extrusion head upon interruption of an extruded filament, or accumulations of extruded filamentary material that may engage and stick to the walls of the attenuation chamber or to previously deposited fiber-forming material.

In effect, one or both of the attenuator sides 16a and 16b "float," i.e., are not held in place by any structure but instead are mounted for a free and easy movement laterally in the direction of the arrows 50 in Figure 1. In a preferred arrangement, the only forces acting on the attenuator sides other than friction and gravity are the biasing force applied by the air cylinders and the internal pressure developed within the attenuation chamber 24. Other clamping means than the air cylinder may be used, such as a spring(s), deformation of an elastic material, or cams; but the air cylinder offers a desired control and variability.

In another useful apparatus of the invention, one or both of the attenuator sides is driven in an oscillating pattern, e.g., by a servomechanical, vibratory or ultrasonic driving device. The rate of oscillation can vary within wide ranges, including, for example, at least rates of 5,000 cycles per minute to 60,000 cycles per second. In still another variation, the movement means takes the form of a negative air pressure (e.g., relative to ambient air pressure) developed within the processing chamber by the venturi air currents.

In the embodiment illustrated in Figures 1-3, the gap width 33 of the attenuation chamber 24 is interrelated with the pressure existing within the chamber, or with the fluid flow rate through the chamber and the fluid temperature. The clamping force matches the pressure within the attenuation chamber and varies depending on the gap width of the attenuation chamber: for a given fluid flow rate, the narrower the gap width, the higher the pressure within the attenuation chamber, and the higher must be the clamping force. Lower clamping forces allow a wider gap width. Mechanical stops, e.g., abutting structure on one or both of the attenuator sides 16a and 16b may be used to assure that minimum or maximum gap widths are maintained.

In one useful arrangement, the air cylinder 43a applies a larger clamping force than the cylinder 43b, e.g., by use in cylinder 43a of a piston of larger diameter than used in cylinder 43b. This difference in force establishes the attenuator side 16b as the side that tends to move most readily when a perturbation occurs during operation. The difference in force is about equal to and compensates for the frictional forces resisting movement of the bearings 38 on the rods 39. Limiting means can be attached to the larger air cylinder 43a to limit movement of the attenuator side 16a toward the attenuator side 16b. One illustrative limiting means, as shown in Figure 3, uses as the air cylinder 43a a double-rod air cylinder, in which the second rod 46 is threaded, extends through a mounting plate 47, and carries a nut 48 which may be adjusted to adjust the position of the air cylinder. Adjustment of the limiting means, e.g., by turning the nut 48, positions the attenuation chamber 24 into alignment with the extrusion head 10.

Because of the described instantaneous separation and reclosing of the attenuator sides 16a and 16b during perturbation of a fiber-forming operation, the operating parameters for the fiber-forming operation are expanded. Some conditions that would previously make the process inoperable -- e.g., because they would lead to filament breakage requiring shutdown for rethreading -- become acceptable with a method and

apparatus of the invention; upon filament breakage, rethreading of the incoming filament end generally occurs automatically. For example, higher velocities that lead to frequent filament breakage may be used. Similarly, narrow gap widths, which cause the air knives to be more focused and to impart more force and greater velocity on filaments passing through the attenuator, may be used. Or filaments may be introduced into the attenuation chamber in a more molten condition, thereby allowing greater control over fiber properties, because the danger of plugging the attenuation chamber is reduced. The attenuator may be moved closer to or further from the extrusion head to control among other things the temperature of the filaments when they enter the attenuation chamber.

Although the chamber walls of the attenuator 16 are shown as generally monolithic structures, they can also take the form of an assemblage of individual parts each mounted for the described floating movement. The individual parts comprising one wall engage one another through sealing means so as to maintain the internal pressure within the processing chamber 24. In a different arrangement, flexible sheets of a material such as rubber or plastic form the walls of the processing chamber 24, whereby the chamber can deform locally upon a localized increase in pressure (e.g., because of a plugging caused by breaking of a single filament or group of filaments). A series or grid of biasing means may engage the flexible wall; sufficient biasing means are used to respond to localized deformations and to bias a deformed portion of the wall back to its undeformed position. Alternatively, a series or grid of oscillating means may engage the flexible wall and oscillate local areas of the wall.

As will be seen, in the preferred embodiment of processing chamber illustrated in Figures 2 and 3, there are no side walls at the ends of the transverse length of the chamber. The result is that fibers passing through the chamber can spread outwardly outside the chamber as they approach the exit of the chamber. Such a spreading can be desirable to widen the mass of fibers collected on the collector. In other embodiments, the processing chamber does include side walls, though a single side wall at one transverse end of the chamber is not attached to both chamber sides 16a and 16b, because attachment to both chamber sides would prevent separation of the sides as discussed above. Instead, a sidewall(s) may be attached to one chamber side and move with that side when and if it moves in response to changes of pressure within the passage. In other embodiments, the side walls are divided, with one portion attached to one chamber side, and the other

portion attached to the other chamber side, with the sidewall portions preferably overlapping if it is desired to confine the stream of processed fibers within the processing chamber.

A wide variety of fiber-forming materials may be used to make fibers with a method and apparatus of the invention. Either organic polymeric materials, or inorganic materials, such as glass or ceramic materials, may be used. While the invention is particularly useful with fiber-forming materials in molten form, other fiber-forming liquids such as solutions or suspensions may also be used. Any fiber-forming organic polymeric materials may be used, including the polymers commonly used in fiber formation such as polyethylene, polypropylene, polyethylene terephthalate, nylon, and urethanes. Some polymers or materials that are more difficult to form into fibers by spunbond or meltblown techniques can be used, including amorphous polymers such as cyclic olefins (which have a high melt viscosity that limits their utility in conventional direct-extrusion techniques), block copolymers, styrene-based polymers, and adhesives (including pressure-sensitive varieties and hot-melt varieties). The specific polymers listed here are examples only, and a wide variety of other polymeric or fiber-forming materials are useful. Interestingly, fiber-forming processes of the invention using molten polymers can often be performed at lower temperatures than traditional direct extrusion techniques, which offers a number of advantages.

Fibers also may be formed from blends of materials, including materials into which certain additives have been blended, such as pigments or dyes. Bicomponent fibers, such as core-sheath or side-by-side bicomponent fibers, may be prepared ("bicomponent" herein includes fibers with more than two components). In addition, different fiber-forming materials may be extruded through different orifices of the extrusion head so as to prepare webs that comprise a mixture of fibers. In other embodiments of the invention other materials are introduced into a stream of fibers prepared according to the invention before or as the fibers are collected so as to prepare a blended web. For example, other staple fibers may be blended in the manner taught in U.S. Patent No. 4,118,531; or particulate material may be introduced and captured within the web in the manner taught in U.S. Patent No. 3,971,373; or microwebs as taught in U.S. Patent No. 4,813,948 may be blended into the webs. Alternatively, fibers prepared by the present invention may be introduced into a stream of other fibers to prepare a blend of fibers.

A fiber-forming process of the invention can be controlled to achieve different effects and different forms of web. For example, a process of the invention can be controlled to control the solidity of filaments entering the processing chamber (e.g., by moving the processing chamber closer to or further from the extrusion head, or increasing or decreasing the volume or the temperature of quenching fluids). In some cases at least a majority of the extruded filaments of fiber-forming material solidify before entering the processing chamber. Such solidification changes the nature of the action of the air impacting the filaments in the processing chamber and the effects within the filaments, and changes the nature of the collected web. In other processes of the invention the process is controlled so that at least a majority of the filaments solidify after they enter the processing chamber, whereupon they may solidify within the chamber or after they exit the chamber. Sometimes the process is controlled so that at least a majority of the filaments or fibers solidify after they are collected, so the fibers are sufficiently molten that when collected they may become adhered at points of fiber intersection.

A wide variety of web properties may be obtained by varying the process. For example, when the fiber-forming material has essentially solidified before it reaches the attenuator, the web will be more lofty and exhibit less or no interfiber bonding. By contrast, when the fiber-forming material is still molten at the time it enters the attenuator, the fibers may still be soft when collected so as to achieve interfiber bonding.

The invention has the advantage that filaments may be processed at very fast velocities not known to be previously available in direct-web-formation processes that use a processing chamber in the same role as the typical role of a processing chamber of the present invention, i.e., to provide primary attenuation of extruded filamentary material. For example, polypropylene is not known to have been processed at apparent filament speeds of 8000 meters per minute in processes that use such a processing chamber, but such apparent filament speeds are possible with the present invention (the term apparent filament speed is used, because the speeds are calculated, e.g., from polymer flow rate, polymer density, and average fiber diameter). Even faster apparent filament speeds have been achieved, e.g., 10,000 meters per minute, or even 14,000 or 18,000 meters per minute, and these speeds can be obtained with a wide range of polymers. In addition, large volumes of polymer can be processed per orifice in the extrusion head, and these large volumes can be processed while at the same time moving extruded filaments at high

velocity. This combination gives rise to a high productivity index -- the rate of polymer throughput (e.g., in grams per orifice per minute) multiplied by the apparent velocity of extruded filaments (e.g., in meters per minute). The process of the invention can be readily practiced with a productivity index of 9000 or higher, even while producing
5 filaments that average 20 micrometers or less in diameter.

Various processes conventionally used as adjuncts to fiber-forming processes may be used in connection with filaments as they enter or exit from the attenuator, such as spraying of finishes or other materials onto the filaments, application of an electrostatic charge to the filaments, application of water mists, etc. In addition, various materials may
10 be added to a collected web, including bonding agents, adhesives, finishes, and other webs or films.

Although there typically is no reason to do so, filaments may be blown from the extrusion head by a primary gaseous stream in the manner of that used in conventional meltblowing operations. Such primary gaseous streams cause an initial attenuation and
15 drawing of the filaments.

The fibers prepared by a method of the invention may range widely in diameter. Microfiber sizes (about 10 micrometers or less in diameter) may be obtained and offer several benefits; but fibers of larger diameter can also be prepared and are useful for certain applications; often the fibers are 20 micrometers or less in diameter. Fibers of
20 circular cross-section are most often prepared, but other cross-sectional shapes may also be used. Depending on the operating parameters chosen, e.g., degree of solidification from the molten state before entering the attenuator, the collected fibers may be rather continuous or essentially discontinuous. The orientation of the polymer chains in the
25 fibers can be influenced by selection of operating parameters, such as degree of solidification of filament entering the attenuator, velocity and temperature of air stream introduced into the attenuator by the air knives, and axial length, gap width and shape (because, for example, shape influences the venturi effect) of the attenuator passage.

Unique fibers and fiber properties, and unique fibrous webs, have been achieved by the invention. For example, in some collected webs, fibers are found that are
30 interrupted, i.e., are broken, or entangled with themselves or other fibers, or otherwise deformed as by engaging a wall of the processing chamber. The fiber segments at the location of the interruption -- i.e., the fiber segments at the point of a fiber break, and the

fiber segments in which an entanglement or deformation occurs -- are all termed an interrupting fiber segment herein, or more commonly for shorthand purposes, are often simply termed "fiber ends": these interrupting fiber segments form the terminus or end of an unaffected length of fiber, even though in the case of entanglements or deformations there often is no actual break or severing of the fiber. The fiber ends have a fiber form (as opposed to a globular shape as sometimes obtained in meltblowing or other previous methods) but are usually enlarged in diameter over the intermediate portions of the fiber; usually they are less than 300 micrometers in diameter. Often, the fiber ends, especially broken ends, have a curly or spiral shape, which causes the ends to entangle with themselves or other fibers. And the fiber ends may be bonded side-by-side with other fibers, e.g., by autogenous coalescing of material of the fiber end with material of an adjacent fiber.

Figure 4 is a scanning electron micrograph at 150X enlargement of a polypropylene fibrous web prepared in Example 5. As seen, the web includes fiber ends 52, which though in fibrous form, have a larger diameter than intermediate or middle portions 53. The interrupting fiber segments, or fiber ends, generally occur in a minor amount. The main portion of the fibers is unaffected (for shorthand purposes, the unaffected main portions of fibers are termed the "middles" herein). Also, the interruptions are isolated and random, i.e., they do not occur in a regular repetitive or predetermined manner.

Fiber ends as described arise because of the unique character of the fiber-forming process of the invention, which continues in spite of breaks and interruptions in individual fiber formation. Such fiber ends may not occur in all collected webs of the invention, but do occur at least at some useful operating process parameters (for example, they may not occur if the extruded filaments of fiber-forming material have reached a high degree of solidification before they enter the processing chamber). Individual fibers may be subject to an interruption, e.g., may break while being drawn in the processing chamber, or may entangle with themselves or another fiber as a result of being deflected from the wall of the processing chamber or as a result of turbulence within the processing chamber, perhaps while still molten: but notwithstanding such interruption, the fiber-forming process of the invention continues. The result is that the collected web includes a significant and detectable number of the fiber ends, or interrupting fiber segments where

there is a discontinuity in the fiber. Since the interruption typically occurs in or after the processing chamber, where the fibers are typically subjected to drawing forces, the fibers are under tension when they break, entangle or deform. The break, or entanglement generally results in an interruption or release of tension allowing the fiber ends to retract and gain in diameter. Also, broken ends are free to move within the fluid currents in the processing chamber, which at least in some cases leads to winding of the ends into a spiral shape and entangling with other fibers.

Analytical study and comparisons of the fiber ends and middle portions, such as the portions 52 and 53 in Figure 4, typically reveals a different morphology between the ends and middles. The polymer chains in the fiber ends usually are oriented, but not to the degree they are oriented in the middle portions of the fibers. This difference in orientation can result in a difference in the proportion of crystallinity and in the kind of crystalline or other morphological structure. And these differences are reflected in different properties.

Figures 5 and 6 present plots obtained by differential scanning calorimetry (DSC) for representative fibers and fiber ends of PET webs prepared in Examples 27 and 29, respectively. The solid-line plots are for intermediate or middle portions of a fiber and the dotted-line plots are for fiber ends. The solid-line plots show a dual melting peak, points 55 and 56 on Figure 5, and points 55' and 56' on Figure 6. The higher-temperature peak, 55 and 55', shows the melting point for a chain-extended, or strain-induced, crystalline portion; and the other peak, 56 and 56', shows the melting point for a non-chain-extended, or less-ordered, crystalline portion. (The term "peak" herein means that portion of a heating curve that is attributable to a single process, e.g., melting of a specific molecular portion of a fiber such as a chain-extended portion; sometimes peaks are sufficiently close to one another that one peak has the appearance of a shoulder of the curve defining the other peak, but they are still regarded as separate peaks, because they represent melting points of distinct molecular fractions.) The existence of a chain-extended crystalline portion generally means that the fibers have superior properties such as tensile strength, durability, and dimensional stability.

From a comparison of the solid-line and dotted-line plots it is seen that in the tested sample the fiber ends, represented by the dotted-line plots, have a lower melting point than the middle portions of the fibers; such a difference in melting point occurs because of a difference between the middles and ends in crystalline structure and orientation. Also, in

the tested sample the fiber ends have a higher cold-crystallization peak (the point 57 and 57' in Figures 5 and 6, respectively; crystallization of an amorphous or semicrystalline material upon heating is called cold crystallization), signifying that the fiber ends contain more amorphous or semicrystalline material, and less highly ordered crystalline material than in the middle portions. The middle portions exhibit cold crystallization, by the peaks 58 and 58', but over a wider and different temperature range than the fiber ends.

A difference in glass transition temperature (T_g) between fiber ends and fiber middles is also often noted during thermal analysis. This difference is more clearly shown in Figures 7 and 7a, which are plots for the middles (solid-line) and ends (dotted-line) for another sample, namely Example 16; Figure 7a is an enlarged view of a portion of the plots on which T_g occurs. The T_g for the middles, point 59, is 9.74 degrees C, while the T_g for the ends, point 60, is - 4.56 degrees C.

In general, when fiber middles and ends prepared by this invention are evaluated using a properly calibrated differential scanning calorimeter (DSC), the fiber middles and ends will differ from each other as to one or more of the common thermal transitions by at least the resolution of the testing instrument (0.1 °C), due to the differences in the mechanisms operating internally within the fiber middles and fiber ends. For example, when experimentally observable, the thermal transitions can differ as follows: 1) the glass transition temperature, T_g , for middles can be slightly higher in temperature than for ends, and the feature can diminish in height as crystalline content or orientation in the fiber middle increases; 2) when observed, the onset temperature of cold crystallization, T_c , and the peak area measured during cold crystallization will be lower for the fiber middle portion relative to the fiber ends, and finally, 3) the melting peak temperature, T_m , for the fiber middles will either be elevated over the T_m observed for the ends, or become complex in nature showing multiple endothermic minima (i.e., multiple melting peaks representing different melting points for different molecular portions that, for example, differ in the order of their crystalline structure), with one molecular portion of the middle portion of the fiber melting at a higher temperature than molecular portions of the fiber ends. Most often, fiber ends and fiber middles differ in one or more of the parameters glass transition temperature, cold crystallization temperature, and melting point by at least 0.5 or 1 degree C.

Webs including fibers with enlarged fibrous ends have the advantage that the fiber ends may comprise a more easily softened material adapted to increase bonding of a web; and the spiral shape can increase coherency of the web.

5

Examples

Apparatus as shown in Figure 1 was used to prepare fibers from a number of different polymers as summarized in Table 1. Specific parts of the apparatus and operating conditions were varied as described below and as also summarized in Table 1. Table 1 also includes a description of characteristics of the fibers prepared.

10

Examples 1-22 and 42-43 were prepared from polypropylene; Examples 1-13 were prepared from a polypropylene having a melt flow index (MFI) of 400 (Exxon 3505G), Example 14 was prepared from polypropylene having a MFI of 30 (Fina 3868), Examples 15-22 were prepared from a polypropylene having a MFI of 70 (Fina 3860), and Examples 42-43 were prepared from a polypropylene having a MFI of 400 (Fina 3960).

15

Polypropylene has a density of 0.91g/cc.

20

Examples 23-32 and 44-46 were prepared from polyethylene terephthalate; Examples 23-26, 29-32 and 44 were prepared from PET having an intrinsic viscosity (IV) of 0.61 (3M 651000), Example 27 was prepared from PET having an IV of 0.36, Example 28 was prepared from PET having an IV of 0.9 (a high-molecular-weight PET useful as a high-tenacity spinning fiber supplied as Crystar 0400 supplied by Dupont Polymers), and Examples 45 and 46 were prepared from PETG (AA45-004 made by Paxon Polymer Company, Baton Rouge, LA). PET has a density of 1.35 and PETG has a density of about 1.30.

25

Examples 33 and 41 were prepared from a nylon 6 polymer (Ultramid PA6 B-3 from BASF) having an MFI of 130 and a density of 1.15. Example 34 was prepared from polystyrene (Crystal PS 3510 supplied by Nova Chemicals) and having an MFI of 15.5 and density of 1.04. Example 35 was prepared from polyurethane (Morton PS-440-200) having a MFI of 37 and density of 1.2. Example 36 was prepared from polyethylene (Dow 6806) having a MFI of 30 and density of 0.95. Example 37 was prepared from a block copolymer comprising 13 percent styrene and 87 percent ethylene butylene copolymer (Shell Kraton G1657) having a MFI of 8 and density of 0.9.

30

Example 38 was a bicomponent core-sheath fiber having a core (89 weight percent) of the polystyrene used in Example 34 and a sheath (11 weight percent) of the

copolymer used in Example 37. Example 39 was a bicomponent side-by-side fiber prepared from polyethylene (Exxact 4023 supplied by Exxon Chemicals having a MFI of 30); 36 weight percent) and a pressure-sensitive adhesive 64 weight percent). The adhesive comprised a terpolymer of 92 weight percent isooctylacrylate, 4 weight percent styrene, and 4 weight percent acrylic acid, had an intrinsic viscosity of 0.63, and was supplied through a Bonnot adhesive extruder.

In Example 40 each fiber was single-component, but fibers of two different polymer compositions were used – the polyethylene used in Example 36 and the polypropylene used in Examples 1-13. The extrusion head had four rows of orifices, with 42 orifices in each row; and the supply to the extrusion head was arranged to supply a different one of the two polymers to adjacent orifices in a row to achieve an A-B-A ... pattern.

In Example 47 a fibrous web was prepared solely from the pressure-sensitive adhesive that was used as one component of bicomponent fibers in Example 39; a Bonnot adhesive extruder was used.

In Examples 42 and 43 the air cylinders used to bias the movable sides or walls of the attenuator were replaced with coil springs. In Example 42, the springs deflected 9.4 millimeters on each side during operation in the example. The spring constant for the spring was 4.38 Newtons/millimeter so the clamping force applied by each spring was 41.1 Newtons. In Example 43, the spring deflected 2.95 millimeters on each side, the spring constant was 4.9 Newtons/millimeter, and the clamping force was 14.4 Newtons.

In Example 44 the extrusion head was a meltblowing die, which had 0.38-millimeter-diameter orifices spaced 1.02 millimeters center to center. The row of orifices was 101.6 millimeters long. Primary meltblowing air at a temperature of 370 degrees C was introduced through a 203-millimeter-wide air knife on each side of the row of orifices at a rate of 0.45 cubic meters per minute (CMM) for the two air knives in combination.

In Example 47 pneumatic rotary ball vibrators oscillating at about 200 cycles per second were connected to each of the movable attenuator sides or walls; the air cylinders remained in place and aligned the attenuator chamber under the extrusion head and were available to return the attenuator sides to their original position in the event a pressure buildup forced the sides apart. During operation of the example, a lesser quantity of pressure-sensitive adhesive stuck onto the attenuator walls when the vibrators were

operating than when they were not operating. In Examples 7 and 37 the clamping force was zero, but sub atmospheric air pressure developed in the processing chamber by venturi air currents returned the moveable side walls to their original position after any perturbations.

5 In each of the examples the polymer formed into fibers was heated to a temperature listed in Table 1 (temperature measured in the extruder 12 near the exit to the pump 13), at which the polymer was molten, and the molten polymer was supplied to the extrusion orifices at a rate as listed in the table. The extrusion head generally had four rows of orifices, but the number of orifices in a row, the diameter of the orifices, and the
10 length-to-diameter ratio of the orifices were varied as listed in the table. In Examples 1-2, 5-7, 14-24, 27, 29-32, 34, and 36-40 each row had 42 orifices, making a total of 168 orifices. In the other examples with the exception of Example 44, each row had 21 orifices, making a total of 84 orifices.

The attenuator parameters were also varied as described in the table, including the
15 air knife gap (the dimension 30 in Figure 2); the attenuator body angle (α in Figure 2); the temperature of the air passed through the attenuator; quench air rate; the clamping pressure and force applied to the attenuator by the air cylinders; the total volume of air passed through the attenuator (given in actual cubic meters per minute, or ACMM; about half of the listed volume was passed through each air knife 32); the gaps at the top and bottom of
20 the attenuator (the dimensions 33 and 34, respectively, in Figure 2); the length of the attenuator chute (dimension 35 in Figure 2); the distance from the exit edge of the die to the attenuator (dimension 17 in Figure 2); and the distance from the attenuator exit to the collector (dimension 21 in Figure 2). The air knife had a transverse length (the direction of the length 25 of the slot in Figure 3) of about 120 millimeters; and the attenuator body
25 28 in which the recess for the air knife was formed had a transverse length of about 152 millimeters. The transverse length of the wall 36 attached to the attenuator body was varied: in Examples 1-5, 8-28, 33-35, and 37-47, the transverse length of the wall was 254 millimeters; in Example 6, 26, 29-32 and 36 it was about 406 millimeters; and in Example
30 7 it was about 152 millimeters.

Properties of the collected fibers are reported including the average fiber diameter, measured from digital images acquired from a scanning electron microscope and using an image analysis program UTHSCSA IMAGE Tool for Windows, version 1.28, from the

University of Texas Health Science Center in San Antonio (copyright 1995-97). The images were used at magnifications of 500 to 1000 times, depending on the size of the fibers.

The apparent filament speed of the collected fibers was calculated from the equation, $V_{\text{apparent}} = 4M/\rho\pi d_f^2$, where

M is the polymer flow rate per orifice in grams/cubic meter,

ρ is the polymer density, and

d_f is the measured average fiber diameter in meters.

The tenacity and elongation to break of the fibers were measured by separating out a single fiber under magnification and mounting the fiber in a paper frame. The fiber was tested for breaking strength by the method outlined in ASTM D3822-90. Eight different fibers were used to determine an average breaking strength and an average elongation to break. Tenacity was calculated from the average breaking strength and the average denier of the fiber calculated from the fiber diameter and polymer density.

Samples were cut from the prepared webs, including portions comprising a fiber end, i.e., a fiber segment in which an interruption taking the form of either a break or an entanglement had occurred, and portions comprising the fiber middle, i.e., the main unaffected portion of the fibers, and the samples were submitted for analysis by differential scanning calorimetry, specifically Modulated DSCTM using a Model 2920 device supplied by TA Instruments Inc, New Castle, DE, and using a heating rate of 4 degrees C/minute, a perturbation amplitude of plus-or-minus 0.636 degrees C, and a period of 60 seconds. Melting points for both the fiber ends and the middles were determined; the maximum melting point peak on the DSC plots for the fiber middles and ends are reported in Table 1.

Although in some cases no difference between middles and ends was detected as to melting point, other differences were often seen even in those examples, such as differences in glass transition temperature.

The samples of fiber middles and ends were also submitted for X-ray diffraction analysis. Data were collected by use of a Bruker microdiffractometer (supplied by Bruker AXS, Inc. Madison, WI), copper K_α radiation, and HI-STAR 2D position sensitive detector registry of the scattered radiation. The diffractometer was fitted with a 300-micrometer collimator and graphite-incident-beam monochromator. The X-ray generator

consisted of a rotating anode surface operated at settings of 50kV and 100mA and using a copper target. Data were collected using a transmission geometry for 60 minutes with the detector centered at 0 degrees (2θ). Samples were corrected for detector sensitivity and spatial irregularities using the Bruker GADDS data analysis software. The corrected data were averaged azimuthally, reduced to x-y pairs of scattering angle (2θ) and intensity values, and subjected to profile fitting by using the data analysis software ORIGINTM (supplied by Microcal Software, Inc. Northhampton, MA) for evaluation of crystallinity.

A gaussian peak shape model was employed to describe the individual crystalline peak and amorphous peak contributions. For some data sets, a single amorphous peak did not adequately account for the total amorphous scattered intensity. In these cases additional broad maxima were employed to fully account for the observed amorphous scattered intensity. Crystallinity indices were calculated as the ratio of crystalline peak area to total scattered peak area (crystalline plus amorphous) within the 6-to-36 degree (2θ) scattering angle range. A value of unity represents 100 percent crystallinity and a value of zero corresponds to a completely amorphous material. Values obtained are reported in Table 1.

As to five examples of webs made from polypropylene, Examples 1, 3, 13, 20 and 22, X-ray analysis revealed a difference between middles and ends in that the ends included a beta crystalline form, measured at 5.5 angstroms.

Draw area ratios were determined by dividing the cross-sectional area of the die orifice by the cross-sectional area of the completed fibers, calculated from the average fiber diameter. Productivity index was also calculated.

Table 1

Example Number	1	2	3	4	5	6	7	8	9	10
Polymer	PP	PP	PP	PP	PP	PP	PP	PP	PP	PP
MFI/IV	400	400	400	400	400	400	400	400	400	400
Melt Temperature (C)	187	188	187	183	188	188	188	188	180	188
Number of Orifices	168	168	84	84	168	168	168	84	84	84
Polymer Flow Rate (g/orifice/min)	1.00	1.00	1.00	1.04	1.00	1.00	1.00	0.49	4.03	1.00
Orifice Diameter (mm)	0.343	0.508	0.889	1.588	0.508	0.508	0.508	0.889	0.889	0.889
Orifice L/D	9.26	6.25	3.57	1.5	6.25	6.25	6.25	3.57	3.57	3.57
Air Knife Gap (mm)	0.762	0.762	0.762	0.762	0.762	0.762	0.762	0.381	1.778	0.381
Attenuator Body Angle (degrees)	30	30	30	30	30	30	30	20	40	20
Attenuator Air Temperature (C)	25	25	25	25	25	25	25	25	25	25
Quench Air Rate (ACMM)	0.44	0.35	0.38	0.38	0.38	0.37	0	0.09	0.59	0.26
Clamping Force (Newtons)	221	221	59.2	63.1	148	237	0	23.7	63.1	43.4
Attenuator Air Volume (ACMM)	2.94	2.07	1.78	1.21	2.59	2.15	2.57	1.06	>3	1.59
Attenuator Gap (Top) (mm)	4.19	3.28	3.81	4.24	3.61	2.03	3.51	2.03	5.33	1.98
Attenuator Gap (Bottom) (mm)	2.79	1.78	2.90	3.07	3.18	1.35	3.51	2.03	4.60	1.88
Chute Length (mm)	152.4	152.4	152.4	152.4	76.2	228.6	25.4	152.4	152.4	152.4
Die to Attenuator Distance (mm)	317.5	317.5	317.5	317.5	317.5	304.8	304.8	304.8	304.8	914.4
Attenuator to Collector Dist (mm)	609.6	609.6	609.6	609.6	609.6	609.6	609.6	609.6	609.6	304.8
Average Fiber Diameter (μ)	10.56	9.54	15.57	14.9	13.09	10.19	11.19	9.9	22.26	14.31
Apparent Filament Speed (m/min)	12600	15400	5770	6530	8200	13500	11200	6940	11400	6830
Tenacity (g/denier)	2.48	4.8	1.41	1.92	2.25	2.58	2.43	2.31	0.967	1.83
Percent elongation to break (%)	180	180	310	230	220	200	140	330	230	220
Draw Area Ratio	1050	2800	3260	11400	1510	2490	2060	8060	1600	3860
Melting Point – Middles (°C)	165.4	165.0	164.1	164.1	165.2	164.0	164.3	165.2	164.3	165.4
Second Peak (°C)										
Melting Point – Ends (°C)	163.9	164.0	163.4	163.4	163.2	162.5	164.0	163.3	164.3	163.2
Second Peak (°C)										
Crystallinity Index – Middles	0.44	0.46	0.42	0.48	0.48	0.52	0.39	0.39	0.50	0.40
Crystallinity Index – Ends	0.56	0.38	0.48		0.4	0.32	0.35	0.34	0.41	0.53
Productivity Index g·m/hole·min ²	12700	15500	5770	6760	8240	13600	11300	3380	45800	6830

Table 1 cont.

Example Number	11	12	13	14	15	16	17	18	19
Polymer	PP	PP	PP	PP	PP	PP	PP	PP	PP
MF/IV	400	400	400	30	70	70	70	70	70
Melt Temperature	190	196	183	216	201	201	208	207	206
Number of Orifices	84	84	84	168	168	168	168	168	168
Polymer Flow Rate	1.00	1.00	1.00	0.50	1.00	0.50	0.50	0.50	0.50
Orifice Diameter	0.889	0.889	1.588	0.508	0.343	0.343	0.343	0.343	0.343
Orifice LD	3.57	3.57	1.5	3.5	9.26	3.5	3.5	3.5	3.5
Air Knife Gap	0.381	1.778	0.762	1.270	0.762	0.762	0.762	0.762	0.762
Attenuator Body Angle	20	40	30	30	30	30	30	30	30
Attenuator Air Temperature	25	25	121	25	25	25	25	25	25
Quench Air Rate	0	0.59	0.34	0.19	0.17	0	0.35	0.26	0.09
Clamping Force	27.6	15.8	55.2	25.6	221	27.6	27.6	27.6	27.6
Attenuator Air Volume	0.86	1.19	1.25	1.24	2.84	0.95	0.95	1.19	1.54
Attenuator Gap (Top)	2.67	6.30	3.99	5.26	4.06	7.67	5.23	3.78	3.78
Attenuator Gap (Bottom)	2.67	6.30	2.84	4.27	2.67	7.67	5.23	3.33	3.33
Chute Length	152.4	76.2	152.4	152.4	152.4	152.4	152.4	152.4	152.4
Die to Attenuator Distance	101.6	127	317.5	1181.1	317.5	108	304.8	292.1	292.1
Attenuator to Collector Dist.	914.4	304.8	609.6	330.2	609.6	990.6	787.4	800.1	800.1
Average Fiber Diameter	18.7	21.98	14.66	16.50	16.18	19.20	17.97	14.95	20.04
Apparent Filament Speed	4000	2900	6510	2570	5370	1900	2170	3350	1740
Tenacity	0.52	0.54	1.68	2.99	2.12	2.13	2.08	2.56	0.87
Percent elongation to break	150	100	110	240	200	500	450	500	370
Draw Area Ratio	2300	1600	12000	950	450	320	360	560	290
Melting Point - Middles	162.3	163.9	164.5	162.7	164.8	164.4	166.2	163.9	164.1
Second Peak				167.3			164.4		
Melting Point - Ends	163.1	163.4	164.3	163.5	163.8	163.7	164.0	163.9	163.9
Second Peak				166.2					
Crystallinity Index - Middles	0.12	0.13	0.46	0.53	0.44	0.33	0.43	0.37	0.49
Crystallinity Index - Ends	0.05	0.42	0.50	0.45	0.43	0.17		0.38	0.44
Productivity Index	4000	2900	6500	1280	5390	950	1080	1680	870
	g·m/hole·min ²								

Table 1 cont.

Example Number	20	21	22	23	24	25	26	27
Polymer	PP	PP	PP	PET	PET	PET	PET	PET
MF/IV	70	70	70	0.61	0.61	0.61	0.61	0.36
Melt Temperature	(°C)	221	221	278	290	281	290	290
Number of Orifices	168	168	168	168	168	84	84	168
Polymer Flow Rate	(g/orifice/min)	0.50	0.50	1.01	1.00	0.99	0.99	1.01
Orifice Diameter	(mm)	0.343	0.343	0.343	0.508	0.889	1.588	0.508
Orifice L/D		3.5	3.5	3.5	3.5	3.57	3.5	3.5
Air Knife Gap	(mm)	0.762	0.762	0.762	1.270	0.762	0.381	1.270
Attenuator Body Angle	(degrees)	30	30	30	30	30	40	30
Attenuator Air Temperature	(°C)	25	25	25	25	25	25	25
Quench Air Rate	(ACMM)	0.09	0.30	0.42	0.48	0.35	0.17	0.22
Clamping Force	(Newtons)	27.6	150	17.0	3.9	63.1	3.9	86.8
Attenuator Air Volume	(ACMM)	1.61	>3	1.61	2.11	2.02	0.64	2.40
Attenuator Gap (Top)	(mm)	3.78	3.78	3.78	4.83	5.08	2.21	5.03
Attenuator Gap (Bottom)	(mm)	3.33	3.35	3.35	4.83	3.66	4.01	3.86
Chute Length	(mm)	152.4	152.4	152.4	76.2	152.4	228.6	152.4
Die to Attenuator Distance	(mm)	508	508	685.8	317.5	317.5	317.5	127
Attenuator to Collector Dist.	(mm)	584.2	584.2	431.8	609.6	609.6	609.6	742.95
Average Fiber Diameter	(μ)	16.58	15.73	21.77	11.86	10.59	13.26	10.05
Apparent Filament Speed	(m/min)	2550	2830	1490	6770	8410	5320	9420
Tenacity	(g/denier)	1.9	1.4	1.2	3.5	5.9	3.6	3.5
Percent elongation to break	(%)	210	220	250	40	30	40	20
Draw Area Ratio		430	480	250	840	2300	5600	2600
Melting Point – Middles	(°C)	165.9	163.9	165.7	260.9	259.9	265.1	256.5
Second Peak	(°C)		167.2		258.5	267.2	----	268.3
Melting Point – Ends	(°C)	164.1	164.0	163.7	257.1	257.2	255.7	257.5
Second Peak	(°C)				253.9	254.3	268.7	----
Crystallinity Index – Middles		0.5	0.39	0.40	0.10	0.20	0.27	0.12
Crystallinity Index – Ends		0.5	0.09	0.51	0	0	0	0
Productivity Index	g·m/hole·min ²	1270	1410	738	6820	8400	5270	9500

Table 1 cont.

Example Number	28	29	30	31	32	33	34	35
Polymer	PET	PET	PET	PET	PET	Nylon	PS	Urethane
MFI/IV	0.85	0.61	0.61	0.61	0.61	130	15.5	37
Melt Temperature	290	282	281	281	281	272	268	217
Number of Orifices	84	168	168	168	168	84	168	84
Polymer Flow Rate	0.98	1.01	1.01	1.01	1.01	1.00	1.00	1.98
Orifice Diameter	1.588	0.508	0.508	0.508	0.508	0.889	0.343	0.889
Orifice L/D	3.57	6.25	6.25	6.25	6.25	6.25	9.26	6.25
Air Knife Gap	0.762	0.762	0.762	0.762	0.762	0.762	0.762	0.762
Attenuator Body Angle	30	30	30	30	30	30	30	30
Attenuator Air Temperature	25	25	25	25	25	25	25	25
Quench Air Rate	0.19	0	0.48	0.48	0.35	0.08	0.21	0
Clamping Force	39.4	82.8	86.8	82.8	82.8	39.4	71.0	86.8
Attenuator Air Volume	1.16	2.16	2.16	2.15	2.15	2.12	2.19	>3
Attenuator Gap (Top)	3.86	3.68	3.68	3.58	3.25	4.29	4.39	4.98
Attenuator Gap (Bottom)	3.10	3.10	3.10	3.10	2.64	3.84	3.10	4.55
Chute Length	762	228.6	228.6	228.6	228.6	76.2	152.4	76.2
Die to Attenuator Distance	317.5	88.9	317.5	457.2	685.8	317.5	317.5	317.5
Attenuator to Collector Distance	609.6	609.6	609.6	482.6	279.4	831.85	609.6	609.6
Average Fiber Diameter	12.64	10.15	10.59	11.93	10.7	12.94	14.35	14.77
Apparent Filament Speed	5800	9230	8480	6690	8310	6610	5940	9640
Tenacity	3.6	3.1	4.7	4.1	5.6	3.8	1.4	3.3
Percent elongation to break	30	20	30	40	40	140	40	140
Draw Area Ratio	16000	2500	2300	1800	2300	4700	570	3600
Melting Point - Middles	268.3	265.6	265.3	262.4	261.4	221.2		23.77
Second Peak	257.3	257.9	269.5		*	218.2		?
Melting Point - Ends	254.1	257.2	257.2	257.4	257.4	219.8		?
Second Peak	268.9	268.4	*	*	*	----	----	----
Crystallinity Index - Middles	0.22	0.09	0.32	0.35	0.35	0.07	0	0
Crystallinity Index - Ends	0	0	0	0	0	<0.05	0	0
Productivity Index	5690	9320	8360	6740	8380	6610	5940	19100
	g-m/hole-min ²							

Table 1 cont.

Example Number	36	37	38	39	40	41	42
Polymer	PE	B1. Copol.	PS/copol.	PE/PSA	PE/PP	Nylon	PP
MFL/IV	30	8	15.5/8	30/63	30/400	130	400
Melt Temperature	(°C)	275	269	205	205	271	206
Number of Orifices		168	168	168	168	84	84
Polymer Flow Rate	(g/orifice/min)	0.99	1.14	0.83	0.64	0.99	2.00
Orifice Diameter	(mm)	0.508	0.508	0.508	0.508	0.889	0.889
Orifice L/D		6.25	6.25	6.25	6.25	6.25	6.25
Air Knife Gap	(mm)	0.762	0.762	0.762	0.762	0.762	0.762
Attenuator Body Angle	(degrees)	30	30	30	30	30	30
Attenuator Air Temperature	(°C)	25	25	25	25	25	25
Quench Air Rate	(ACMM)	0.16	0.34	0.25	0.34	0.34	0.33
Clamping Force	(Newtons)	205	0.0	27.6	23.7	150	41.1
Attenuator Air Volume	(ACMM)	2.62	0.41	0.92	0.54	2.39	>3
Attenuator Gap (Top)	(mm)	3.20	7.62	3.94	4.78	4.19	3.25
Attenuator Gap (Bottom)	(mm)	2.49	7.19	3.56	4.78	3.76	2.95
Chute Length	(mm)	228.6	76.2	76.2	76.2	76.2	76.2
Die to Attenuator Distance	(mm)	317.5	666.75	317.5	330.2	539.75	317.5
Attenuator to Collector Dist	(mm)	609.6	330.2	800.1	533.4	590.55	609.6
Average Fiber Diameter	(μ)	8.17	34.37	19.35	32.34	12.8	16.57
Apparent Filament Speed	(m/min)	19800	771	4700	1170	6700	10200
Tenacity	(lb/dtex)	1.2		1.2	1.1	3.5	0.8
Percent elongation to break	(%)	60		30	100	50	170
Draw Area Ratio		3900	220	690	250	4800	2900
Melting Point - Middles	(°C)	118.7					165.1
Second Peak	(°C)	123.6					
Melting Point - Ends	(°C)	122.1					164.5
Second Peak	(°C)						
Crystallinity Index - Middles		0.72		0	0	0.36	0.43
Crystallinity Index - Ends		0.48		0	0	0.26	0.47
Productivity Index	g-m/hole-min ²	19535	497	5340	972	7040	20400

Table 1 cont.

Example Number	43	44	45	46	47
Polymer	PP	PET	PETG	PETG	PSA
MFI/IV	400	0.61	>70	>70	0.63
Melt Temperature (°C)	205	290	262	265	200
Number of Orifices	84	**	84	84	84
Polymer Flow Rate (g/orifice/min)	2.00	0.82	1.48	1.48	0.60
Orifice Diameter (mm)	0.889	0.38	1.588	1.588	0.508
Orifice L/D	6.25	6.8	3.5	3.5	3.5
Air Knife Gap (mm)	0.762	0.762	0.762	0.762	0.762
Attenuator Body Angle (degrees)	30	30	30	30	30
Attenuator Air Temperature (°C)	25	25	25	25	25
Quench Air Rate (ACMM)	0.33	0	0.21	0.21	0
Clamping Force (Newtons)	14.4	98.6	39.4	27.6	***
Attenuator Air Volume (ACMM)	2.20	1.5	0.84	0.99	0.56
Attenuator Gap (Top) (mm)	4.14	4.75	3.66	3.56	6.30
Attenuator Gap (Bottom) (mm)	3.61	4.45	3.38	3.40	5.31
Chute Length (mm)	76.2	76.2	76.2	76.2	76.2
Die to Attenuator Distance (mm)	317.5	102	317	635	330
Attenuator to Collector Distance (mm)	609.6	838	610	495	572
Average Fiber Diameter (μ)	13.42	8.72	19.37	21.98	38.51
Apparent Filament Speed (m/min)	15500	10200	3860	3000	545
Tenacity (g/denier)	3.6	2.1	1.64	3.19	----
Percent elongation to break (%)	130	40	60	80	----
Draw Area Ratio	4388	1909	6716	5216	1699
Melting Point - Middles (°C)	164.8	257.4			
Second Peak (°C)		254.4			
Melting Point - Ends (°C)	164.0	257.4			
Second Peak (°C)		254.3			
Crystallinity Index - Middles	0.46	<0.05	0	0	
Crystallinity Index - Ends	0.41	0	0	0	
Productivity Index g-m/hole-min ²	31100	8440	5700	4420	330

* multiple values

**meltblowing die,

*** walls oscillated at 200 cycles/sec.

What is claimed is:

1. A method for making fibers comprising a) extruding filaments of fiber-forming material; b) directing the filaments through a processing chamber defined by two parallel walls, at least one of the walls being instantaneously movable toward and away from the other wall and being subject to movement means for providing instantaneous movement during passage of the filaments; and c) collecting the processed filaments.

2. A method of claim 1 further characterized in that the movement means comprises biasing means for resiliently biasing the at least one movable wall toward the other wall, the biasing means providing a biasing force that establishes a dynamic equilibrium between the pressure within the processing chamber and the biasing force such that the wall moves away from the other wall in response to increases in pressure within the chamber but is quickly returned to the equilibrium position by the biasing force upon resumption of the original pressure within the chamber.

3. A method of claim 1 further characterized in that the movement means comprises oscillating means for oscillating the at least one movable wall at a rapid rate so as to release extrudate that may accumulate on the walls of the chamber.

4. A method of claim 1 in which both parallel walls are instantaneously movable toward and away from one another and subject to movement means for providing instantaneous movement.

5. A method of claim 1 in which a fluid stream is established to direct the filaments through the processing chamber, and at least part of the fluid stream flows through narrow slots disposed within the processing chamber and has a vector component along the longitudinal axis through the processing chamber.

6. A method of claim 1 in which the parallel walls have a length transverse to the direction of filament movement through the chamber substantially greater than the spacing between the walls.

7. A method of claim 6 in which the processing chamber is free of side walls at the ends of the transverse length of the parallel walls.

5 8. A method of claim 1 controlled so that at least a majority of the filaments solidify before entering the processing chamber, whereupon the solidified filaments are subjected to a lengthwise orienting stress within the chamber.

10 9. A method of claim 1 controlled so that at least a majority of the filaments solidify after they enter the processing chamber but before they exit the chamber.

10. A method of claim 1 controlled so that at least a majority of the fibers solidify after they exit the processing chamber.

15 11. A method of claim 1 controlled so that at least a majority of the fibers are sufficiently liquid when collected that fibers become adhered at points of fiber intersection.

20 12. A method of claim 1 in which fibers are collected at an apparent filament speed of at least 8000 meters per minute.

25 13. A method of claim 1 in which the fiber-forming material is extruded through a plurality of die orifices arranged side-by-side in at least one row, and the individual filaments are attenuated into microfibers having an average fiber diameter of about 10 micrometers or less.

30 14. A method for making fibers comprising a) extruding filaments of fiber-forming liquid through orifices in a die, b) directing the filaments through an attenuation chamber defined by two parallel walls, at least one of the walls being instantaneously movable toward and away from the other wall and being resiliently biased toward the other wall; c) establishing a fluid stream that carries the filaments between the walls and attenuates them into fibers; d) selecting a biasing force on the at least one movable wall

that establishes a dynamic equilibrium between the pressure within the attenuation chamber and the biasing force such that the wall moves away from the other wall in response to increases in pressure within the chamber but is quickly returned to the equilibrium position by the biasing force upon resumption of the original pressure within the chamber; and e) collecting the formed fibers.

15. A method of claim 14 in which both parallel walls are instantaneously movable toward and away from one another and connected to biasing means for providing such instantaneous movement.

16. Apparatus for forming fibers comprising a) an extrusion head for extruding filaments of fiber-forming material through orifices in a die, b) a processing chamber aligned to receive the extruded filaments for passage through the chamber, the chamber being defined by two parallel walls, at least one of the walls being instantaneously movable toward and away from the other wall; and c) movement means for providing instantaneous movement of at least one wall.

17. Apparatus of claim 16 in which the movement means comprises biasing means for resiliently biasing the wall toward the other wall, movement of the wall toward and away from the other wall being sufficiently easy and rapid that the wall will move away from the other wall in response to increases in pressure within the chamber but will be quickly returned to its original position by the biasing means upon resumption of the original pressure within the chamber.

18. Apparatus of claim 17 in which the biasing means comprises an air cylinder having a sliding piston connected to said at least one wall, and the pressure applied to the piston can be adjusted to adjust the force biasing said wall toward the other wall.

19. Apparatus of claim 16 in which the movement means comprises oscillating means for oscillating the wall at a rapid rate.

20. Apparatus of claim 16 in which both parallel walls are instantaneously movable toward and away from one another and connected to movement means for providing instantaneous movement.

5 21. Apparatus of claim 16 which further includes air knives disposed within the processing chamber and providing a fluid stream having a vector component along the longitudinal axis of the processing chamber to increase the velocity of filaments passing through the chamber.

10 22. Apparatus of claim 16 in which the at least one movable wall is segmented into parts that can move individually toward and away from the other wall.

23. Apparatus of claim 16 in which the width of the chamber is narrower near the entry to the chamber than toward the exit of the chamber.

15 24. A nonwoven web comprising a collected mass of fibers that includes fibers randomly interrupted by isolated fiber segments that comprise oriented polymer chains but differ in morphology from the main portion of the fiber.

20 25. A nonwoven web of claim 24 in which the difference in morphology is represented by a difference in at least melting point, cold-crystallization temperature, or glass transition temperature, as measured by differential scanning calorimetry, or crystallinity index or a difference in crystallinity type as measured by X-ray scattering.

25 26. A nonwoven web of claim 24 in which the interrupting segment is fiber-like as opposed to globular and is less than 300 micrometers in diameter, but larger in diameter than the main portions of the fiber.

30 27. A nonwoven web of claim 24 in which the interrupting fiber segment comprises a broken end of the interrupted fiber.

28. A nonwoven web of claim 24 in which the interrupting fiber segment comprises an entanglement of the interrupted fiber with itself or another fiber.

5 29. A nonwoven web of claim 24 in which the interrupted fibers have an average diameter of about 10 micrometers or less.

10 30. A nonwoven web of claim 24 in which the main portion of the interrupted fiber exhibits multiple melting peaks during differential scanning calorimetry, with the different melting peaks representing molecular fractions within the fiber that differ in degree of crystalline order.

31. A nonwoven web of claim 24 in which the main portion of the interrupted fiber exhibits a dual melting peak during differential scanning calorimetry, with one melting peak representing a chain-extended crystalline portion.

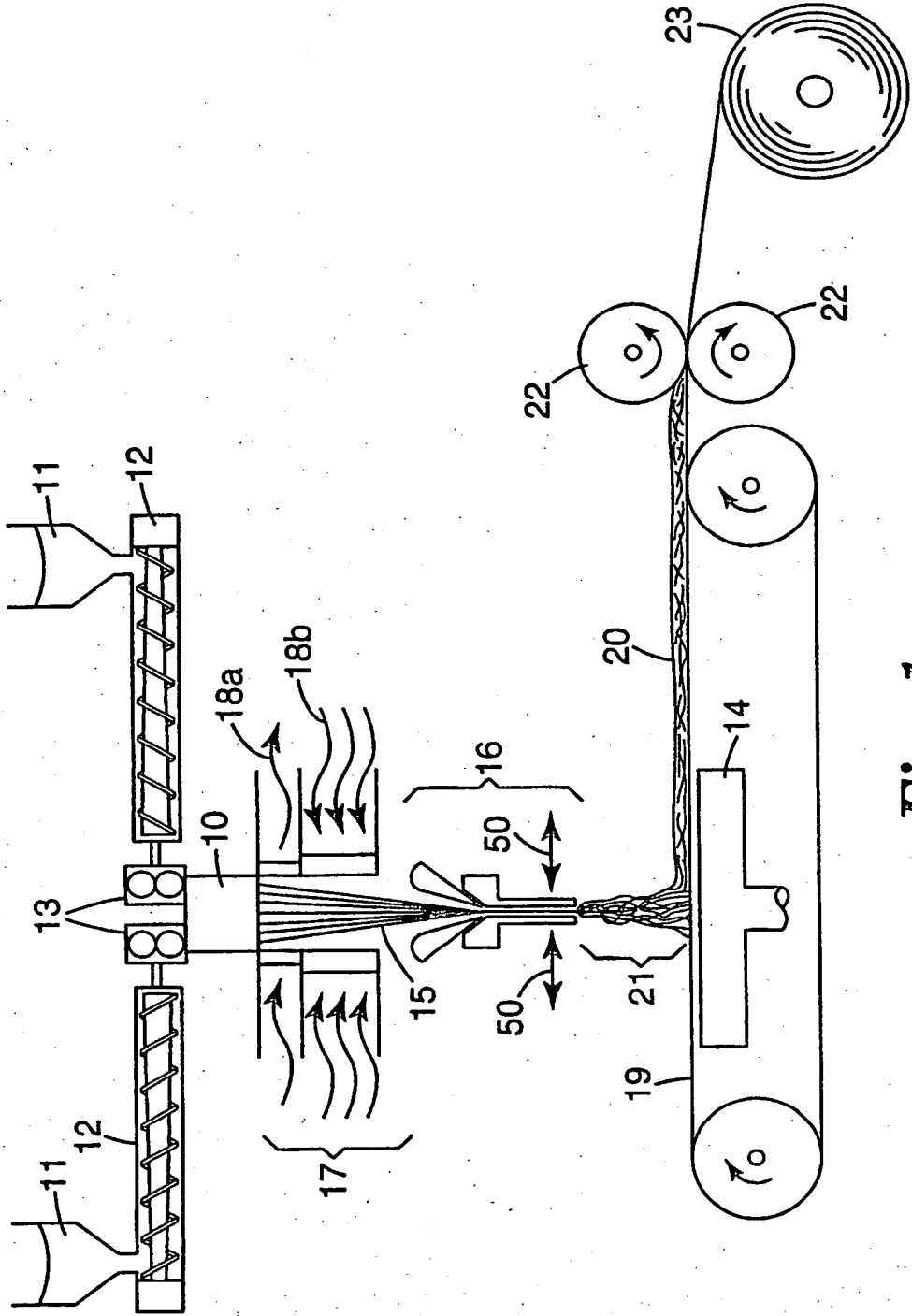
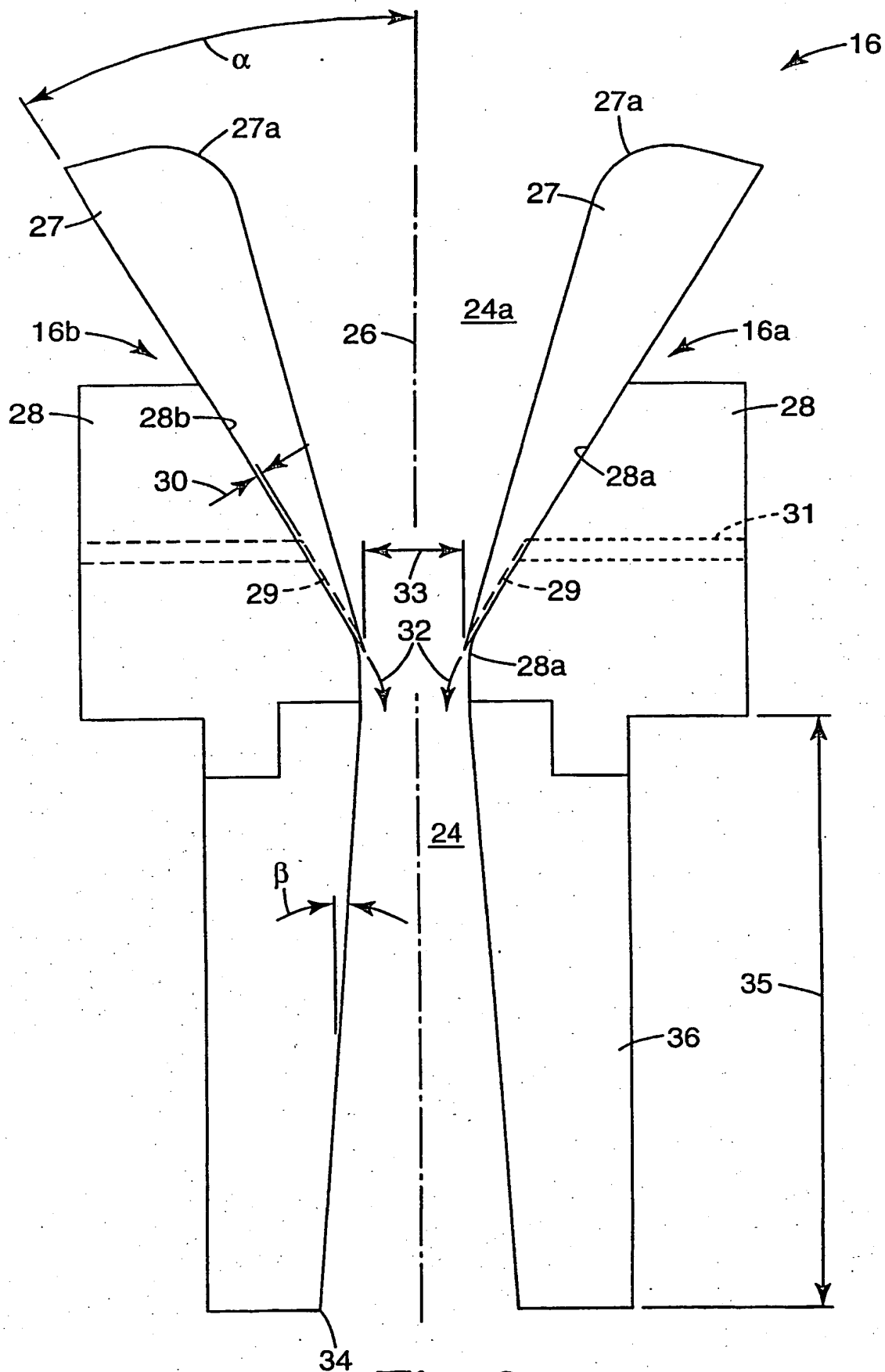
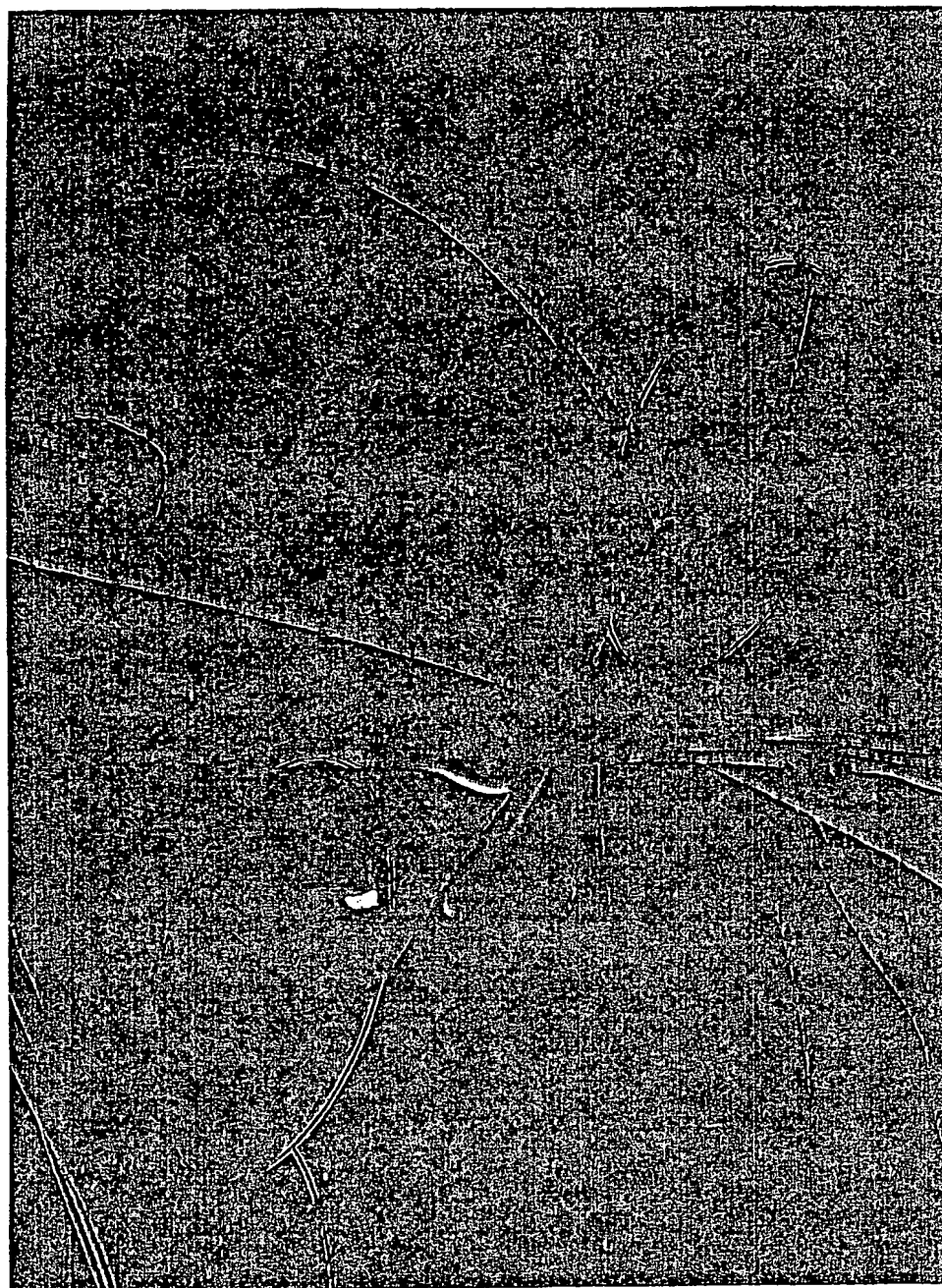


Fig. 1

**Fig. 2**

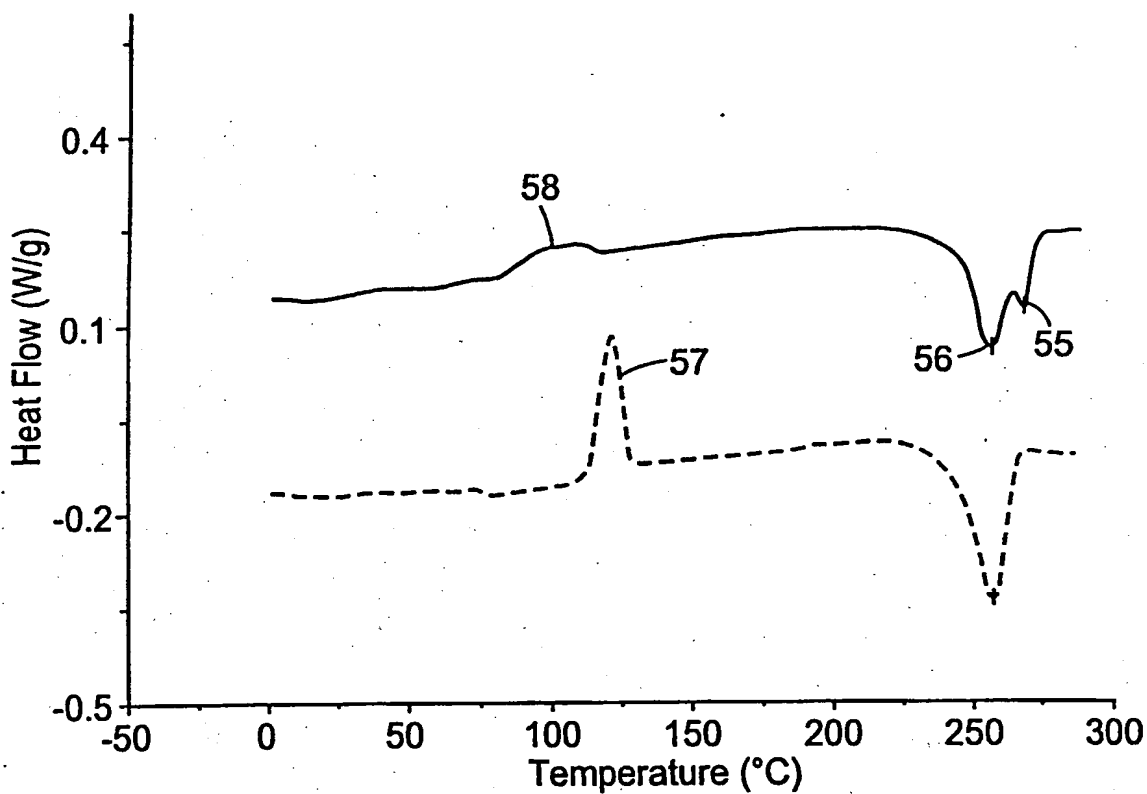
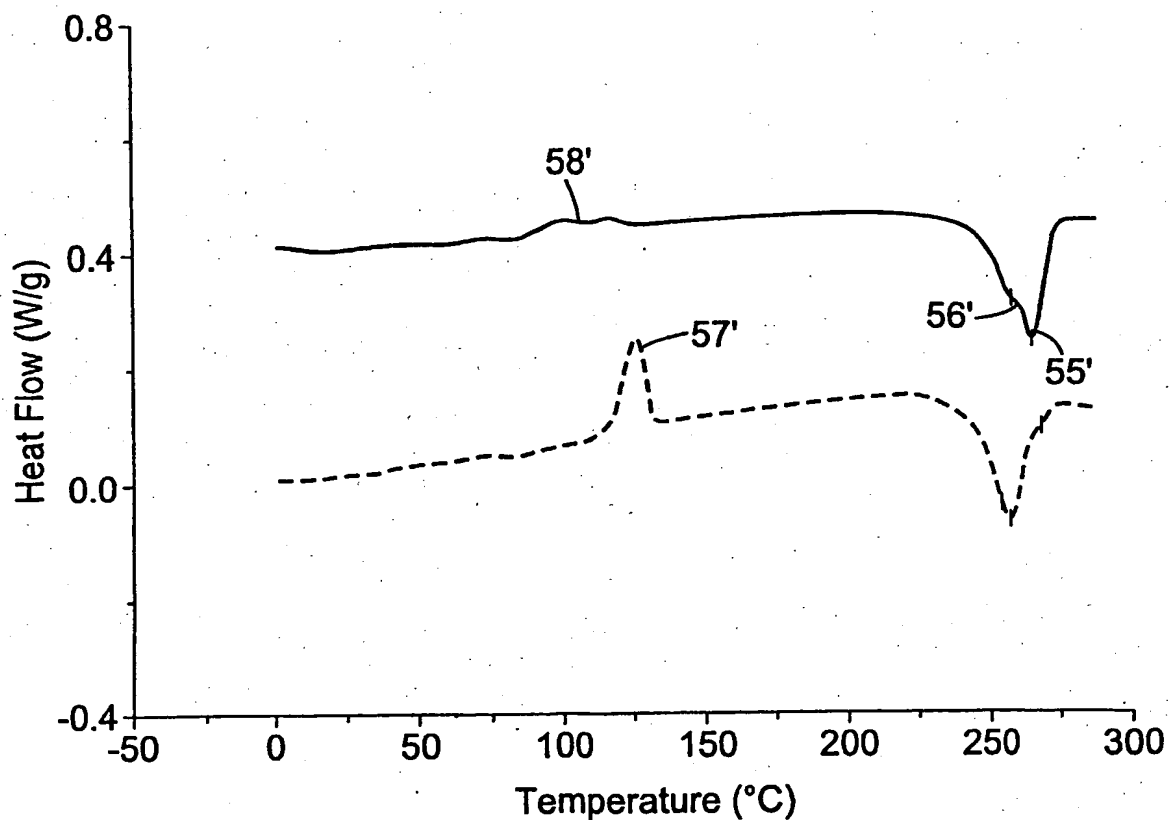
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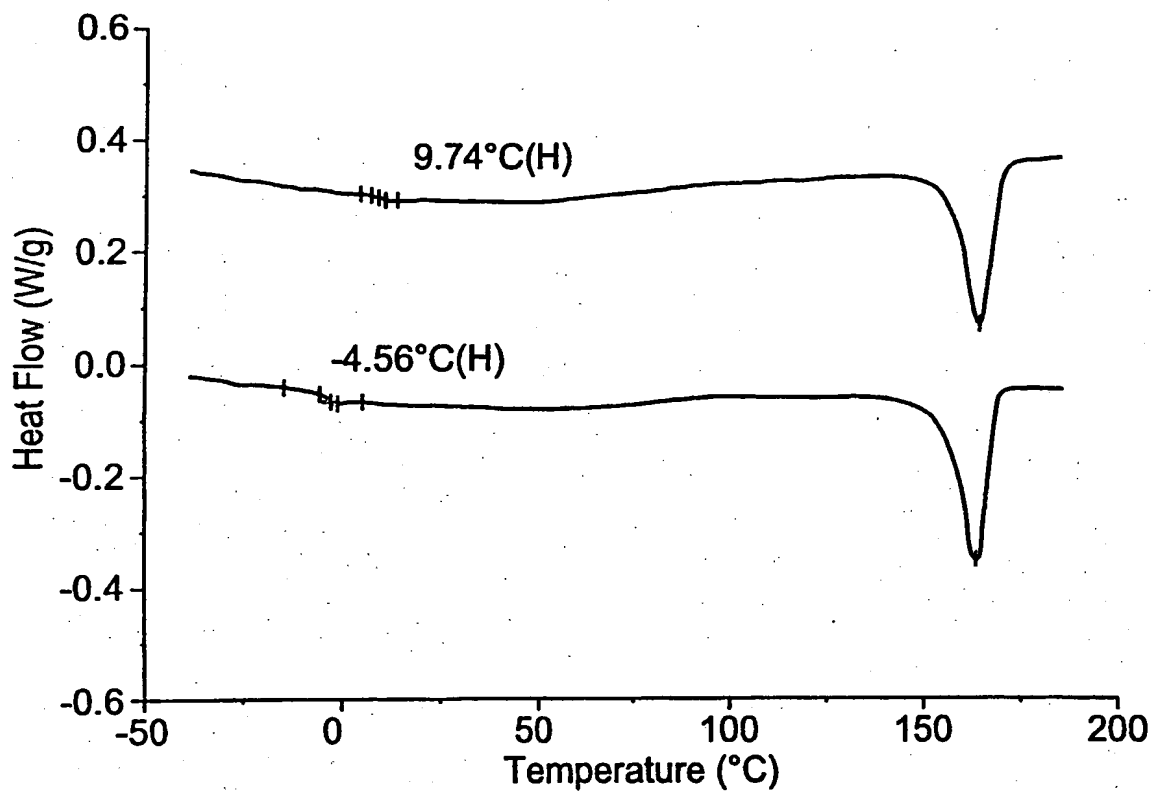
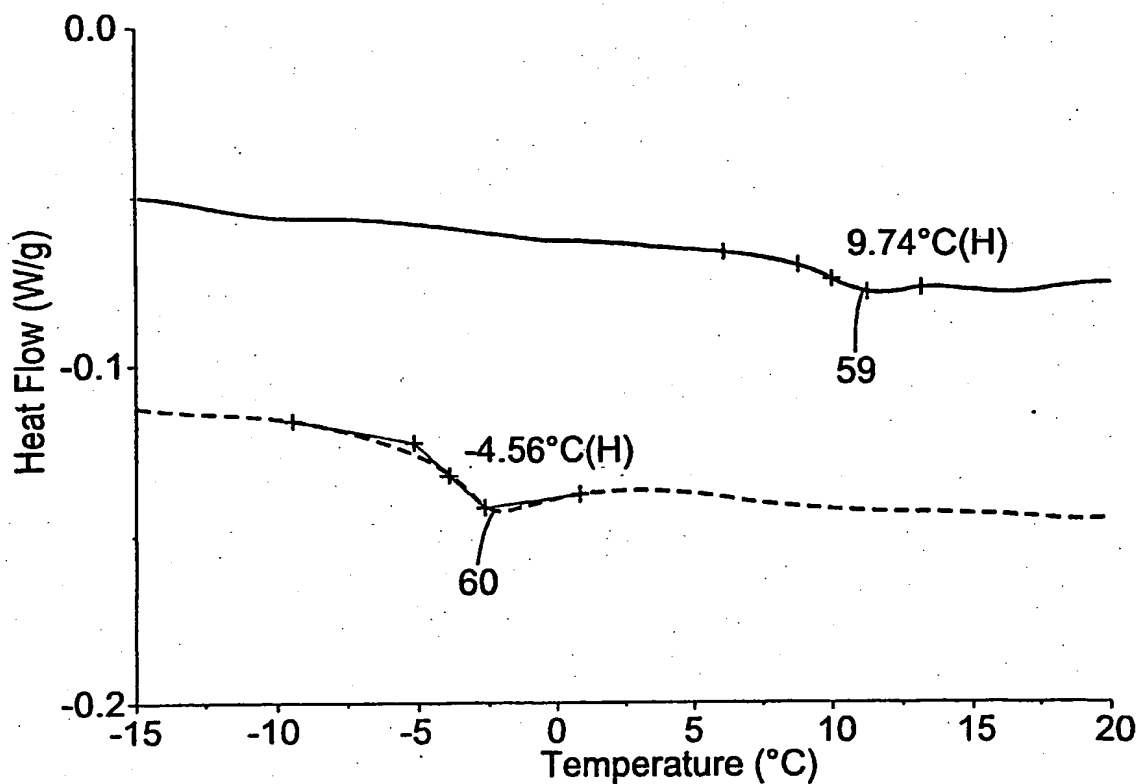
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Fig. 4

*Fig. 5**Fig. 6*

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*Fig. 7**Fig. 7a*

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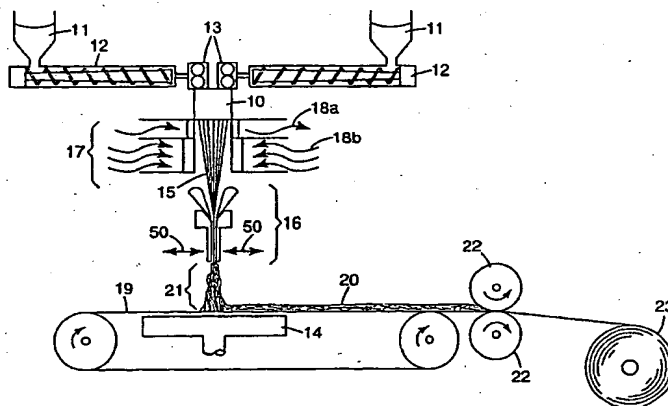
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[Continued on next page]

(54) Title: FIBER-FORMING PROCESS



(57) Abstract: A new fiber-forming method, and related apparatus, are taught in which extruded filaments of fiber-forming material are directed through a processing chamber that is defined by two parallel walls, at least one of which is instantaneously movable toward and away from the other wall; preferably both walls are instantaneously movable toward and away from one another. Movement means are connected to the at least one movable wall to provide instantaneous movement. In one embodiment, the movement means comprises biasing means for resiliently biasing the wall toward the other wall. Movement of the wall toward and away from the other wall is sufficiently easy and rapid that the wall will move away from the other wall in response to increases in pressure within the chamber but will be quickly returned to its original position by the biasing means upon resumption of the original pressure within the chamber. In another embodiment the movement means comprises oscillating means for oscillating the wall at a rapid rate. The invention also provides new nonwoven webs, which comprise a collected mass of fibers that includes fibers randomly interrupted by isolated fiber segments that comprise oriented polymer chains but differ in morphology from the main portion of the fiber.

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According to International Patent Classification (IPC) or to both national classification and IPC

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Minimum documentation searched (classification system followed by classification symbols)

IPC 7 D01D D04H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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